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Deinking of Paper Using a Water Solvent Emulsion System

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DEINKING OF PAPER USING A WATER SOLVENT EMULSION SYSTEM

by

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ABSTRACT

This study examines the use of a solvent and water emulsion to deink waste paper. Solvents looked at in detail included naphthalene and paradichlorobenzene. Various grades of waste paper were tested for deinkability including, coated and heavily printed magazine cover stock, plastic coated SBS board, beer case stock with hotmelts, uncoated and unfilled stock printed with a neutral black ink, old corrigating medium filled with hotmelts and stickies, and finally, board printed with UV-cured inks. Most of these samples deinked very well.

Brightness and appearance were the basis for measuring deinkability. Yield was also tabulated for selected experiments. As a spin-off from these experiments it was possible to consider the use of these solvents in a 100% solvent deinking system.

INTRODUCTION

Current trends show that paper recycling has increased moderately over the past twenty years.[8,15] With this in mind, as well as predicted tightening in the pulp supply, one must look critically at every possible method of ink removal. Solvents have been used since the 1930's to deink paper.[1] More recently its been found that solvents are useful to remove plastics, hotmelts, resinous material, and glues from paper.[1,9] This study will examine the possibility of combining the best of an aqueous system with the advantages of a solvent system. Chlorinated hydrocarbons have already been looked at for this purpose.[1] A recent literature search indicates that nobody had looked at plastics dissolved in a suitable solvent, paradichlorobenzene, or napthalene. Furthermore, these solvents have never been studied for their use in a 100% solvent deinking system, such as the Riverside or Polysolv process. Interestingly enough however, these compounds are either floatable or possess the unique chemical property of being easily sublimated.[12]

THEORETICAL DISCUSSION

Much of todays waste paper is considered to be useless because conventional methods of deinking simply cannot remove many of todays coatings. This problem has been growing every year. Todays waste paper supply has been contaminated with variable amounts of plastics, hotmelts, glues and stickies, latexs, and ultraviolet cured inks.

Presently we have four basic methods of ink removal from paper. These methods are washing, flotation, solvent extraction, and screw extracting.[5,6]

The advantages of a washing deinking system are; 1) Inorganic filler can be removed or controlled, as well as fines content, 2) Can adjust easier to variations in wastepaper input quality, and 3) Lower capital investment and chemical costs compared to flotation deinking.

The disadvantages of the system include; 1) Lower yield than the flotation method, 2) Higher water consumption and therefore larger quanities of contaminated waste water that must be treated. Typically, this system uses the following pulping chemicals; sodium hydroxide at levels of 2-5% based on the fiber input, 2-6% of sodium silicate based on the fiber also and, 0.5-1% surfactant based on the fiber to help wet the fibers.[5,6] The Surfactant helps to wet the fibers, as stated above, thereby allowing or inhancing the action of the alkali. The alkali tends to wet and swell the fibers but in the process the fibers become weakened.[6] Because of this, sodium silicate is added to reduce the harshness of the pulping and to help reduce discoloration of groundwood grades.

The advantages of the flotation deinking system are; 1) The ink

is removed in a concentrated form, thus decreasing water treatment, 2) Yields as high as 85-95% of the input wastepaper weight are reported, 3) Because of the lower water consumption it is easier to close the system and 4) It is adventageous to use with some light-weight contaminants.[5,6]

Some of the disadvantages to the flotation method include; 1) Fillers and fines are not removed, 2) Higher capital and chemical costs, and 3) More sensitive to upsets and variations in feed.[5,6]

It is common for a flotation cell to run at 0.9-1.2% consistancy at temperatures of 104-129°F for eight to ten minutes duration. Various chemicals used inpulping and deinking, include, sodium peroxide (2%), sodium silicate (4%) foamer (0-0.25%) and a collector (0.03-0.8%). In the pulper the pH is greater than 9.5 and in the flotation cell the pH is 8-9. The sodium peroxide is equivalent to sodium hydroxide and hydrogen peroxide in a water medium, therefore it had the dual purpose of swelling the fibers and bleaching them as well.

The collector chemicals used in the flotation cell are made of fatty-acid soaps and promote foam or bubble stability. The foamer chemicals act on the same principle but are generally composed of detergents or other surfactants. The sodium silicate in this process acts as a dispersing and penetrating agent while it also stabilizes the peroxide.

The screw extraction process has a dilution factor of 32.3 and only requires 6,970 gallons of water to deink a ton of paper. This is nearly four times less water requirement compared to the Sidehill screen.[5]

The Riverside solvent extraction process is of particular

interest to this study because of its similarities in many respects to the combination water and solvent deinking system talked about in this paper. The advantages of the solvent system are unique, as well as, highly cost effective. These advantages are; 1) No contact with water system, thus, no fiber loss, no water treatment and no fiber degradation due to harsh deinking chemicals, 2) Works on many inks as well as common contaminants, such as, wax, polyethylene and other plastics, asphalt, urea and melamine formaldehyde resins, most hotmelts in general and, other latexs and adhesives,[2,6,9] and 3) No changes in the deinking solvent with grade changes. Only temperature changes are made.[16] 4) Plastics and waxes can be recovered and sold[2,16], 5) Waste materials are recovered practically solvent free for disposal[9], 6) Nearly 100% of the solvent is recovered and that portion that isn't may be burned with the waste plastics or waxes being removed from the paper[16], 7) Essentially, the process is pollution free [2], and 8) The current cost to deink a ton of paper may be as low as $40.00 per ton in some cases.[2] The disadvantages of this system are virtually non-existent. It must be born in mind however, that solvents are expensive and toxic. Flash points of a given solvent as well as vapor toxicity must be considered also. A variety of chemicals have been looked at as being potentially good deinking solvents. For example, the Riverside Corporation uses perchloroethylene. Tom Mestetsky and B.G. Webster in conjunction with G.A.F. Corporation have a patent on N-alkyllactam as a deinking solvent, and other solvents such as, trichloethylene, orthodichlorobenzne & cyclohexane have been tried, to name but a few.[1,20] In the "Polysolv Process" or the Riverside process we also find that at higher temperature the wet strength resins are softened

which lends itself to more efficient subsequent pulping of the reclaimed fiber.[10] It was also shown in laboratory tests that there was no strength loss between solvent extracted and non-extracted fiber.[10]

The repulpability of waste paper is determined by the temperature, time, pH, consistancy and the wetability of the paper.[11,18] This is true to a large extent for solvent systems and emulsion systems. Furthermore, in an emulsion system, the ink particle size and, contact time of the solvent and ink become critical.

Inks used in printed material are composed of pigments, dyes, vehicles, drying agents, and gloss agents. The vehicle tends to bind a given pigment to a substrate material. In turn the drying agents and gloss agents affect or modify the vehicle and therefore the final print. Vehicles are generally composed of oil modified alkydes, oleoresinous varnishes, and rosin esters or phenolic modified resin esters in a suitable solvent.[3,17] Although the composition varies widely depending upon the method of printing to be employed, the type of printing press to be used, and the speed of the press. The dyes used in inks are composed of a wide range of organic compounds, such as, azo dyes, stilbene dyes and xanthene dyes.[3]

There is a broad spectrum of waste paper that is recognized as being difficult or impossible to deink in a conventional water system. Some of these papers are listed below, 1) Those containing hotmelts or stickies, 2) Latexs, 3) Plastics, 4) Varnishes, and 5) Ultraviolet-cured inks.[11] One should notice however, that most if not all of these "problem papers" can be dealt with effectively using a solvent system.

Solvents are currently being used in many of the nations industries.

They are used as a medium for chemical reactions, as a vehicle in paints, in lacquers in printing inks, as cleaners, and for purification of substances. If water is excluded as a solvent then the coating industry is the largest user of chemical solvents.[19] A solvent is defined by one author as, "any substance capable of dissolving other substances to form a homogeneous system called a solution". Another definition of a solvent is, "a system of two or more components in a single phase".[19] Solvents can be made up of a single chemical compound or, have a multicomponent composition.

An emulsion is, "an intimate mixture of liquids, one of which (the disperse phase) is distributed in large or small globules throughout the other (the continuous phase)".[19] An emulsifying agent, without which the emulsion would break down is generally present at the two phase interface. The properties of an emulsion depend on the interaction of the following factors; 1) the emulsion type, 2) the concentration of each phase, 3) the degree of dispersion and, the nature of the emulsifying agent. The stability of an emulsion is dependent upon the size of the globules and, the nature of their surfaces.[7,13,19] These factors are influenced by, 1) the length of time an emulsion settles, 2) the temperature at which the emulsion is kept, 3) the amount of mechanical or electrical energy put into the suspension, 4) the pH of the system, 5) the order in which components of the emulsion are added as well as their concentrations and, 6) finally, the type of emulsifying agents used. Globules will coalescence. The emulsifying agent acts in one of four ways to stabilize the globules; 1) by supplying negatively charged ions for adsorption on the globules, 2) by surrounding the globules with a film of molecules, such as, a protein, a soap or

a detergent, 3) by coating the globules with certain solid particulates and, 4) emulsions can also be stabilized unnaturally by the use of force, either mechanical or electrical.[7,13] Emulsions can be formed under certain conditions with water and trichloroethylene, water and napthalene, water and paradichlorobenzene, and water mixed with styrofoam dissolved in dimethyl ether.[14,21] In each of these cases the water represents the continuous phase while the solvent is the dispersed phase. All of the above solvents are immissible in water and tend to form layers in water if not mixed.

Napthalene or tar camphor is the most abundant constituent in coal tar, comprising about 11% of its dry total.[12] It sublimes appreciably at temperature above its melting point, and is volatile in steam. The flash point of this compound in and open cup is 79°C and 88°C in a closed cup.[12] It's autoignition temperature is 567°C.[12] Napthalene had a molecular weight of 128.18, a melting point of 82.2°C, a boiling point of 217.9°C, and a density of 1.145gm/cc in its solid state. As a liquid its density is less than water. Currently, it is used to manufacture phthalic acid, anthranilic acids, sulfonic acids, and related compounds used in the dye industry. Napthalene is also used in the manufacture of synthetic resins, celluloid, and lampblack.[12]

Paradichlorobenzene is similar to napthalene in many ways. Orthodichlorobenzene has been recognized as an excellent solvent, and when crystals of paradichlobenzene are melted they also have excellent solvent properties. The crystals are volatile and can be sublimed at ordinary temperatures. The melting point is between 53°and 54°C depending on the molecular configuration, and its boiling point is 174.12°C.[12] It is non-corrosive. The flash point of

this compound is 150°F in a closed cup.[12] Paradichlorobenzene has a molecular weight of 147.01 and a density of 1.533 . Neither napthalene nor paradichlobenzene are soluble in water.

Styrofoam has a density of 1.05gm/cc in its pure form, and much less when air is mixed into it to form the common styrofoam cup. When diethyl ether is mixed with styrofoam the styrofoam dissolves and produces a lighter than water solvent mixture. The density of ether is 0.7079gm/ml at 25°C.[12] Other plastics could be prepared in a similar fashion but they aren't as readily available as strofoam or as easily dissolved in common solvents.

Experimental Introduction

This study is based upon eleven different experiments and a mathematical analysis of data. For each experiment a section on experimental procedure, data, and discussion is included. The procedure sections cover emulsion deinking, and solvent deinking, as well as, other related deinking procedures. Included on the next page is a list of experiments to aid the reader. For more information concerning the mathematical techniques used in this artical, see appendix "A".

LIST OF EXPERIMENTS

I. Eutectic formed from camphor and napthalene--the decreases in melting points are examined. See graph 1-A.

II. Emulsion of water and napthalene used to deink.

III. Napthalene used as a deinking solvent without water.

IV. Combination solvent of napthalene and trichloroethylene used to deink.

V. Emulsion of water and paradichlorobenzene used to deink.

VI. Paradichlorobenzene used as a deinking solvent without water.

VII. Emulsion of styrofoam, diethyl ether, and trichloroethylene.

VIII. Napthalene and paradichlorobenzene, comparison of deinking using only solvent at constant temperatures.

IX. The effects of chemical concentration variations on the deinkability of napthalene. See graph 1-B.

X. Emulsion deinking using water and napthalene solvent. Trials were run on various inks, hotmelts and stickies.

XI. Emulsion of napthalene and water used to deink magazine cover stock. Dupont experimental analysis was used on this data to determine the relationship between the blending time, the temperature of the reaction, and the chemical concentration.

EXPERIMENTAL PROCEDURE I.

This experiment examines the melting points of nine different combinations of napthalene and camphor. Reasons for examination of the melting points was to determine whether or not small additions of camphor could be used to reduce the solvents' melting point, and thus save energy costs for deinking.

A Mettler P160N balance was used to weigh samples of napthalene and camphor to the nearest 0.01 gram. For each of the eutectic combinations 1 gram samples were prepared. The melting points of the eutectics were measured using a heated water bath and a cappilary tube filled with the crystaline mixture.

This tube was attached to a standard 76mm glass bulb thermometer using a small rubber band. The thermometer scale was marked every 1mm for each 1^{o} centigrade. Both the thermometer and tube were lowered into a water bath made from a 2 liter beaker. The bath was stirred and heated very slowly till the first sample melted. The rest of the melting points were determined using the same procedure. In each case the temperature was approximated to the nearest 0.1^{o} centigrade.

Data I.

The melting points of the following eutectic combinations were taken, and recorded in the following table.

Data I.

Table 1-A

Napthalene (%)	Camphor (%)	Melting point (°C)
100	0	80.2
95	5	78.5
90	10	77.0
80	20	71.0
70	30	67.0
60	40	57.0
50	50	48.0
40	60	34.5
20	80	31.0

Table 1-A: Note; See graph of this relationship at end of article.

Discussion (I.)

This experiment reveled that additions of 20% camphor were needed to decrease the temperature of the napthalene solvent by ten degrees centigrade. The addition of this much camphor raised the cost of the solvent by 44%. Furthermore, camphor is slightly soluable in water, and this portion would not be recoverable from the deinking process. As indicated by these facts, it would not be economical to use camphor to lower the melting point of napthalene.

EXPERIMENTAL PROCEDURE II.

This experiment examines the use of a napthalene-in-water emulsion to deink a printed and, plastic coated stock (Munch Peanut bar box). Prior to running this experiment, five test runs were made to help in setting up a useful deinking procedure. It was determined that the one quart size Waring blender pulped best at about 3% consistancy. Therefore, in all of the 5 and 10 gram runs the consistancy used was 3.3%. Each run was done by itself and took between 45 minutes and $1^1/4$ hours from start to finish.

The preliminary procedure included heating the blender container and the buchner funnel with hot distilled water to prevent crystalization of the napthalene. An emulsion was formed by adding 5 grams of napthalene into 150ml of distilled water at 85°C, and allowing it to melt. Into a Waring blender 5 grams of box board and 0.1 grams of TSPP were added followed by the heated water-napthalene solvent. The mixture was pulped in the "blend" mode for 30 seconds and then dilluted with 500ml of 85°C distilled water. The floating ink, napthalene and fiber were skimmed from the liquid surface and discarded. The slurry was then "flash blended" for a second or two and the floating material was removed as before. This procedure was repeated four more times. Approximately 150ml of waste material was removed and 150ml of fresh 85°C water added. Each time less ink was skimmed off the surface. Finally, a pad was collected in a buchner funnel and dried on a hotplate.

Data II.

The dryed pad was weighed and its brightness measured using an Elrepho brightness meter. A yield of 26.8% was calculated by weight. The average of ten brightness values, five to each side, was 74.0. The pad obtained from this experiment was as soft as tissue paper, and easily ripped. Some small specks of ink did get into the pad in some trial runs.

Discussion II.

An all glass deinking apparatus was necessary because of napthalene ability to dissolve or soften many common plastics. Due to the simplistic design of the deinking cell it was nearly impossible to control the yield and remove the floating ink simultaneously. It was noticed that less fiber floated after it was in the emulsion-water for more than 15 minutes. It's expected that the wetability of the fiber had something to do with this phenomenon. The Elrepho brightness meter was used in all of these experiments because it makes use of an integrating sphere which gives a better brightness when measuring rough surfaces. The average Elrepho brightness for the base stock was (77.2) brightness units. The brightness of the basestock was an average of ten points taken from the backside of the printed board. This surface was uncoated and devoid of print. This corresponded to a brightness loss of (3.2%) due to both the deinking process and the residual ink after deinking. Some of this brightness loss is also attributed to slightly over-drying the pad. The yellow ink was the most retained color. Small, barely visible specks were distributed throughout the pad. More ink could have easily been removed if the "blend & skim" deinking process was continued.

EXPERIMENTAL PROCEDURE III.

This experiment will briefly examine the use of 100% napthalene as a deinking solvent. A test sample of slightly over seven grams of plastic coated (Munch Peanut bar box) board was dipped into 85°C napthalene to observe the solvent effect. This experiment was done only once. However, the ink and plastic were removed so well that another run would have been redundant. First, a heated water bath was prepared in which a tared 2 liter beaker was placed containing 167.6 grams of napthalene. After the 85°C temperature was reached, the sheet of board was dipped intact into the napthalene solvent. Within 10 to 15 seconds the plastic coat and print were totally removed from the sheet. The board was then taken out of the solvent and allowed to dry. This sheet was weighed to get a general idea of the yield out of the solvent bath. Finally, the board was cut into one inch squares to be pulped. The sample was added to a pre-heated one quart size Waring blender with 300ml of 85°C distilled water and 0.1 grams of TSPP. The mixture was blended to a pulp using the "blend", button for 30 seconds, and filtered in a 15cm buchner funnel. The filtered pad was dried on a hotplate and a final weight was taken.

Data III.

After deinking the beaker was reweighed and the total solvent and ink weight was 158.9 grams. The original board sample was 7.1707 grams and after deinking its weight was 6.3200 grams, a drop in weight of less than 12%. The pad weight after pulping and filtering was 6.0122 grams. We observed that the solvent turned pink as the board was deinked. Average brightness of the pads was 67.4, as

measured on an Elrepho brightness meter.

Discussion III.

One problem was encountered during this experiment. Namely, that there were substantial losses of napthalene due to sublimation, thus, eliminating the possibility of a material balance. Crystals of napthalene could be seen floating up and over the edge of the beaker. The small decrease in fiber after pulping was due in part to fiber loss in the blender. The final pad was completely void of ink specks. The yield was at least 88% of the input weight. The brightness of the pad (67.4) was lower than the pad of the previous experiment because of excessive scorching during drying. It's the authors opinion that if the pad hadn't been scorched it would have had a brightness comparable to the pad obtained in experiment II. In this experiment we found that napthalene is an excellent solvent for removal of the plastic and ink from board. It was also noticed that the sublimation properties of napthalene may make it attractive in a 100% napthalene deinking system.

EXPERIMENTAL PROCEDURE IV.

This experiment was a continuation of original efforts to lower the solvent temperature, and save energy costs. A saturated solution of napthalene was prepared by adding 160 grams of napthalene to 300ml of trichloroethylene at 25°C, or at room temperature. The solvent mixture was heated to various temperatures and the inked plastic coated board was dipped in the solvent till the coat and ink were removed. Temperatures of 65°, 47°, 32°, and 22°C were used in this experiment. With one run being done for each of the mentioned temperatures.

Data IV.

The following information was collected for the various solvent temperatures. In every case the ink and plastic films were removed completely.

Solvent Temperature (°C)	Ink removal time (sec.)
1) 65°C	15 seconds or less
2) 47°C	15-20 seconds
3) 32°C	20-25 seconds
4) 22°C	30-40 seconds

Table 1-B

Discussion IV.

It was observed that the samples deinked in less time at elevated temperatures. A comparison between the solvent properties of napthalene compared to napthalene and trichloroethylene was not done. However, trichloroethylene is an excellent deinking solvent by itself.[6] Further examination of this aspect could be done. However, chlorinated

hydrocarbons use has been restrained in recent years due to the potential toxicity in the work place.[6]

EXPERIMENTAL PROCEDURE V.

An attempt was made in this experiment to deink a sample sheet using a heavier than water solvent. In a 250 ml beaker 150 ml of distilled water was heated to 60°C and 5 grams paradichlorobenzene was added. In a one quart Waring blender 5 grams of Home Center magazine cover stock was added along with 0.1 grams of TSPP. The water-paradichlorobenzene mixture was then poured in and the cover stock defibered in the "blend" mode for 30 seconds. Another 500 ml of 65°C distilled water was added to the blender and the mixture was "flash blended" for a second or two. Floating ink was skimmed off the top of the slurry as was done in Experiment II. See appendix "A" for additional information. Finally, the mixture was poured through a 15cm buchner funnel under a vacume to remove water.

Data V.

The pad that formed in the funnel was low in brightness 64.8 and yield 32.8%. The paper was multicolored with spots of blue green, and yellow. Large flecks of colored material were retained in the pad. These inked particles ranged in size from barely visible with the naked eye to $\frac{1}{4}$ inch in diameter. Some of the colored material was very dark blue and the general color of the pad was light blue. The pad color was two-sided, one side being much lighter than the other.

Discussion V.

As expected, the deinking ability of paradichlorobenzene in a

water slurry was very poor. Apparently however, ink was being removed to some extent by the solvent action of the chemical as evidenced by the two-sided pad color. Although this compound was looked at in an emulsion system it's expected to have little practical use as such. The low yield was due in part to the poor wetability of this paper sample. It should also be mentioned that yield was never a primary concern of this study but rather the ability of an emulsion system to remove ink, at any cost.

EXPERIMENTAL PROCEDURE VI

This experiment examines the use of 100% paradichlorobenzene to deink two different base stocks. In a 100ml beaker, 20 grams of paradichlorobenzene were weighed, and melted on a hotplate. Once the solvent temperature reached 60°C the sample was dipped into the solution for 15 seconds. Upon removal from the solvent, the sample was rubbed with a paper towel to remove excess solvent and ink.

DATA VI

The solvent removed the plastic and ink quickly, and completely. It took approximately 13 seconds to deink this board thoughly. The magazine cover stock did not deink nearly as well. After 15 seconds in the paradichlorobenzene solvent, this test sample changed to a purple color. Indicating that dyes were being liberated from the film.

DISCUSSION VI

One of the important aspects of this experiment was the fact that pigments as well as dyes were released from the magazine cover stock. Furthermore, we found that the more plastic-like or highly polymerized the coating was, the more easily it was removed. Of particular interest is the fact that paradichlorobenzene deinks well at a lower temperature than napthalene. Even though it could not be used in a water emulsion, it may be useful in a pure solvent system.

EXPERIMENTAL PROCEDURE VII

An emulsion of plastic and a suitable solvent was prepared, and used to deink a paper sample. The solvent used in this experiment consisted of 10ml of diethyl ether, 1 gram of strofoam, and 1 ml of trichloroethylene. The solvent was prepared and used at 22^{o} centigrade. Only 2 grams of Home Center magazine cover were used in this experiment. The paper was cut into one inch square pieces and added to a dry one quart size Waring blender. One-tenth of a gram of TSPP was also added as a dispersing agent. Ten milliliters of prepared solvent were added to 150ml of 22^{o}C distilled water, and poured over the contents in the blender. The contents were then blended for 30 seconds using the "blend" mode.

The paper sample was defibered well after blending, however, very little ink floated to the surface of the slurry mixture. These results were expected because a brief test was done by dipping a one inch strip of magazine cover stock into the solvent mixture and then rubbing it with paper toweling. We observed that the solvents ability to solubilize the magazine printing was limited compared to napthalene or paradichlorobenzene.

After pulping was completed an additional 300ml of 22^{o}C distilled water was added to the blender, and floating material was skimmed off the top of the mixture using a small beaker. The contents were "flash blended" and skimmed two more times. Approximately 150ml of floating material and emulsion were removed from the slurry surface. A pad was formed in a 15cm buchner funnel and dryed on a hotplate. The experiment was run only once because of the poor deinking done while using this solvent mixture.

Data VII.

The prepared solvent was cloudy-white and floated on water at 22° centigrade. The filtered pad was evenly dispersed with inked particles. These particles ranged in size from barely visible to slightly larger than 1/8 inch in diameter. An unusual difference was noticed in the pad texture when compared with the pads formed from the napthalene or paradichlorobenzene deinking experiments. The pad surface was rough and felt like sandpaper. Upon touching the surface, small, hard, bluish white particles rubbed off.

Discussion VII.

At the onset of preparing this solvent there were no intentions of using trichloroethylene in the mixture. Especially since the EPA had become interested in reducing their use in water systems.[6] However, the ether and styrofoam did not produce a homogenious solution. Instead, the ether melted the styrofoam and they formed two distinct layers. The addition of the trichoroethylene dispersed the styrofoam and ether into each other, thereby producing a true solution. It was expected that the solvent mixture could be used to collect ink and float it to the surface of the deinking cell. However, this was not the case. Instead, it appeared that the plastic solution indiscriminately attached itself to fiber as well as ink particles. Other plastic solution-solvents might deink quite well, and should be examined.

EXPERIMENTAL PROCEDURE VIII

This experiment was run to examine the solvent properties of napthalene compared with those of paradichlorobenzene. First, a one liter heated water bath was prepared using a 2 liter beaker and a Dylatherm 500 watt hotplate. One inch wide strips of unfilled and uncoated paper, plastic coated box board, and magazine cover stock were cut into 6 inch strips. Two 50 ml beakers were suspended in the heated water, and 20 grams of napthalene was placed in one while bath temperature was held constant at 85°C. One end of each test strip was dipped into the 85°C napthalene for 15 seconds, and rubbed using paper toweling. The same procedure was repeated on the other end of each strip using paradichlorobenzene.

Data VIII.

The plastic coated board (Munch peanut bar) was thoroughly deinked and uncoated after treatment with either napthalene or paradichlorobenzene. The magazine cover stock (Home Center magazine) was also deinked by both solvents, but not as completely as the plastic coated sample. The last paper sample was unfilled and uncoated. It was printed with a neutral black ink, and contained no toner dyes. Both attempts to remove ink from this sample resulted in minimal success. It was noticed however, that slightly more ink was removed while using napthalene on this sample.

Discussion VIII.

This experiment shed some light on the types of paper that could be deinked using napthalene or paradichlorobenzene. The best ink removal

was noticed when the printed film on the paper was not "soaked into" the surface. Therefore, the plastic coated board was deinked or "cleaned" the best while the coated magazine stock was deinked less well. Finally, the ink that was soaked into the sheet the most deinked or solubilized the least. The only explaination for napthalene removing more ink must have to do with the chemistry of the ink in relation to the solvent being used. This area could be studied in greater depth.

EXPERIMENTAL PROCEDURE IX.

This experiment considers the effects of changing the concentration of napthalene in a water emulsion deinking system. Five, 10 gram samples of plastic coated board (Munch peanut bar box) were weighed to the nearest hundredths of a gram using a Mettler P160N balance. The napthalene was weighed into 2,5,7, and 10 gram amounts. In each of the five runs 0.1 grams of TSPP was used as a dispersing agent. In each run the paper board was cut into one inch square pieces and placed in a heated one quart Waring blender. The TSPP was then added. Each of the napthalene samples were mixed with 300ml of 85°C distilled water and, allowed to melt. This mixture was then poured over the contents in the blender. The contents were mixed in the "blend" mode for 30 seconds each. The pulp was transferred from the blender to a 2 liter beaker, and 1800 ml of 85°C distilled water was added to dilute the slurry. The liquid level was maintained by addition of 150 ml of 85°C distilled water for each 150 ml of floating ink and napthalene removed. In each run a glass stirring rod was used to promote flotation of the ink, plastic and solvent. As the waste material floated to the surface with napthalene, it was skimmed off using a small beaker. Four 150 ml beakers of waste ink, plastic and napthalene were removed and discarded for each solvent run. The control run could not be skimmed because the ink and plastic did not separate from the pulp slurry. After the skimming was completed for each run the deinked pulp slurry was vacume filtered in a 15cm buchner funnel. The pad was then dried on a hotplate. Once the pads were dry an Elepro brightness was taken, and a visual inspection made. Finally, yields were calculated for all five runs.

Data IX,

The table below records the Elrepro brightness, the yield, color, and overall appearance of the deinked pad from this experiment.

Napthalene	Brightness	Pulp Color	Appearance	Yield
0 gram	73.2	White	Large plastic flecks	96.3
2 gram	73.6	Very light yellow	Few small flecks	50.4
5 gram	73.7	Slightly yellow	Few small flecks	51.9
7 gram	65.4	Slightly golden yellow	Few small flecks	45.6
10 gram	68.0	Light yellow	Very few small flecks	58.6

It was observed that there were losses of napthalene from the surface of the slurry via sublimation. The amount lost in each case was assumed to be constant although this probably isn't the case. For the 7 gram run the temperature went up to 93°C before it was discovered and corrected. All other runs were held at 85°C $\pm$ 2°C using a hotplate. In all the pads there was an observable odor of napthalene present, even the control pad.

Discussion IX.

The amount of lost napthalene may or may not have affected the results of this experiment. However, the loss of napthalene through sublimation poses a potential method for recovery of this particular solvent. The 7 gram run attained higher temperatures than any other run and as a result the pad color was a darker shade of yellow. In all

cases, except the control sample, there was a large decrease in plastic and ink material left in the pad. Interestingly enough, the quantity of inked particles left in the deinked pads was about the same for each sample. There was also a decrease in the pad brightness at chemical concentrations above 5 grams of napthalene. The yellow color was least pronounced in the 2 gram sample. Furthermore, in each sample, (excluding the 7 gram run), the yellow color became progressively more intense. This information supports the conclusions that less than a 20% chemical concentration may substantially remove ink from this particular board sample, and the higher the napthalene concentration the greater the quantity of dye put into solution. It's believed that the increased amounts of dye in solution produced increasingly yellow pads.

EXPERIMENTAL PROCEDURE X.

In this experiment a napthalene-in-water emulsion is used to deink various paper and board samples, while holding the chemical concentration, the blend time, temperature, the volume of floating waste removed, the amount of additives, and the weight of the paper sample constant. This experiment was run using the following grades of waste paper and board;

1) Beer case- varnish and stickies present
2) Old corrugating medium- hotmelts and stickies
3) Uncoated and unfilled sheet- neutral black ink
4) UV-cure inked box- UV-ink

In each case the one quart Waring blender was preheated with boiling water to prevent crystallization of the napthalene, as well as, heat loss from the deinking mixture. Four 2 liter beakers were filled with distilled water, and heated until they boiled using a Lindberg hotplate. Meanwhile, 10 gram samples of the above waste papers were weighed out using a Mettler P160N balance. In each experiment 10 grams of napthalene was weighed to the nearest one-hundredth of a gram just prior to use, so as to prevent unnecessary sublimation losses. Each of the paper samples were cut into one inch squares to facilitate blending. In every run 0.1 grams of TSPP was added as a dispersing agent.

The beer case (Strch's beer case) was the first sample to be run. All other samples were done using the same basic format. The 10 grams of napthalene was added to 300ml of boiling water. The board sample and the TSPP were placed inside the blender. As soon as the napthalene melted the solvent-water mixture was poured over the contents inside the blender. It was necessary to use a variable transformer to start each of the blending periods, because of rapid gas expansion and

spattering from the blender at these high temperatures. The blender speed was gradually increased from the off position to the "blend" mode within a 10 second time span. After the contents were in the "blend" mode for 30 seconds the blender was turned off. Another 300 ml of boiling 98°C distilled water was added and the contents were mixed using the "flash blend" button for a second or two. The contents were then emptied into a 2 liter beaker and placed on a hotplate. More boiling distilled water was added to dilute the slurry to a total volume of 2 liters or about ½% consistancy. The mixture was stirred using a glass rod, and floating material was skimmed off the liquid surface. The stir and skim procedure was repeated until four 150 ml beakers were filled with rejected material. As each beaker was filled with ink and solvent waste another 150 ml of boiling distilled water was added to maintain the liquid level. After the deinking procedure was completed the beaker's contents were filtered through a buchner funnel and the pad was dried on a hotplate.

Data X.

At the higher deinking temperatures used in this set of experiments there was a substantial loss of napthalene due to sublimation on the liquid surface.

The liquid portion of the pulp slurry from the beer case was milky-pink in color. Upon closer examination of this liquid it was observed that the milky-pink particles appeared to form part of a colloidal system. The filtered pad was clean and free from all ink, stickies and hotmelts. The yield was 53.4%.

Upon deinking the old corrugating medium a film formed on the surface that was crystalline in appearance. This film was grey-black

in color. A color that closely resembled that of the hotmelts and stickies material present in the unpulped sheet. The deinked pad was free from any noticeable hotmelts or stickies. The yield obtained from this run was 80.3%.

Pulping of the paper printed with neutral black ink produced a bluish grey liquid, however, material still floated to the surface and was removed by skimming. The pad that was produced after deinking was blue-grey colored. The colored pad appeared as if the fibers had been dyed. There were also minute particles of ink present in the final pad. The Elrepro brightness of the pad was 58.4%, with a yield of 91.7%.

Pulping of the UV-cure inked box produced a slight yellow colored solution. This seemed unusual because red, green, blue and black UV-inks were present in the unpulped board. This stock did not deink perfectly. The pad produced after deinking contained some unpulped material, as well as, some fibers that had been yellowed during the process. The yellowed fibers tended to congregate on the bottom side of the pad. The pad brightness was 75.2, and the yield was 37.4%.

For a complete tabulation of data from this experiment, as well as that of experiment II the reader is directed to the graph and table section of this paper. The table (page44), includes the type of paper deinked using a napthalene and water emulsion, the amount of ink removal, percent yield, base stock brightness, pad brightness, and which experiments the sample pads came from.

Discussion X.

At 98°C the sublimation rate was greatly increased compared with the sublimation rate at 85°C. Therefore, even more problems developed while trying to contain the solvent. Napthalene crystals formed in the

cool air above the opened beaker deinking cell, and deposited around the circumference of the vessel. It would be difficult to estimate the amount of solvent lost through sublimation. However, the amount should be about the same for all runs.

Stroh's Beer Box Sample

The milky-pink color of the pulping liquid could be attributed to several things. 1) A colloid formed from the napthalene and varnish. 2) The formation of a colloid from the napthalene and the hotmelts present on the sample of board we pulped, or 3) One of the above combinations being stabilized by the TSPP in the pulping liquid. The yield was not very impressive. Although, it was comparable with what we obtained in experiment IX using similar ink removal methods.

Old Corrugating Medium Sample

The old corrugating medium was quite different from the other paper and board sample used throughout these experiments. There was no ink present on this paper except that which had attached itself to the hotmelts and stickies found in the sheet. The reasons for using the napthalene emulsion for removal of hotmelts and stickies are twofold. First, it was observed that the napthalene dissolved plastic coating present on one type of board sample. Second, we noticed that napthalene helped to float the ink and plastic, so it could be removed and discarded. See experiment II for additional details. The author believes that the napthalene acted as a collector in this experiment, thus the crystalline appearance on the surface of the deinking slurry. This is not to say that the napthalene didn't also dissolve some of the hotmelts and stickies. The yield was much better for this particular run, however, it was not a controlled

variable in any of the deinking experiments.

Unfilled and Uncoated Paper Sample

In this run the napthalene may have reacted with the compounds present in the ink to produce the blue gray color in the pulp pad. Another possibility is that the ink particles may have formed a colloid with the napthalene and TSPP. In either case the pad was uniformly colored purple-grey, and considerable brightness was lost. However, it may be possible to increase the brightness of the pulp by conventional bleaching methods.

UV-cure Inked Box

Prior to the actual deinking of the UV-cure inked box, a small amount of melted napthalene was rubbed on a UV-inked surface. All colors were present, including red, green, yellow, blue, and black. Each was solubilized and rubbed off.

The reason for some of the fibers turning more yellow than others, and congregating on the bottom side of the filter pad would require a more detailed examination of the pad and inks present. The low yield of 37.4% may be due in part to the greater difficulty encountered in pulping this board. The idea being that the larger, unpulped pieces of board tended to be more difficult to wet, and therefore floated during the deinking operation.

EXPERIMENTAL PROCEDURE XI.

In this experiment we will examine the effects on deinking of magazine cover stock (Home Center Magazine), while using two levels of addition for each of three factors. The basic design is n-factorial(2^3) in nature, as outlined by the Dupont experimental method which it follows.[22] The two levels consist of a high value and a low value for each of the factors. The factors are the temperature (X), blend time (Y), and the napthalene concentration (Z). The following table outlines the conditions for each of the runs.

Trial Run	Temperature (^{o}C)	Blend Time (sec)	Napthalene (g)
1	82	20	0
2	82	20	5
3	82	45	0
4	82	45	5
5	98+	20	0
6	98+	20	5
7	98+	45	0
8	98+	45	5

For each trial condition 5 grams of magazine cover stock and 0.1 grams of TSPP were used. The paper and TSPP was added to a heated, one Quart Waring blender. Then a mixture of 5 grams of napthalene in 150ml of hot distilled water or simply 150 ml of hot distilled water was poured over the blender contents. At the higher temperatures it was necessary to use a variable transformer to increase the blend speed gradually, and prevent spattering. However, the transformer was used for both temperatures to decrease experimental error in the results. After blending for the prescribed time the resultant slurry was poured into a a 2000 ml beaker and dilluted with 1800 ml of hot distilled water. The mixture was then stirred using a glass rod to promote flotation of the ink, and napthalene. In between stirring the liquid, the floating

material was skimmed off. The stir-skim procedure was repeated until four 150 ml beakers had been filled and discarded. In between the removal of each 150 ml of floating material an equivalent volume of hot distilled water was added. The deinked pulp was filtered using a suction flash and a 15cm buchner funnel, and the pads were air dried.

Data XI.

After the pads had been left in a constant temperature and constant humidity room overnight, ten Elrepro brightnesses were taken on each pad. Five to each side. The resultant data is tabulated below.

Trial No.	Temp.	Bl. Time	Napth.	Elrepro Br. Average		Range of Individual determinations
				Top	Bottom	
1	82	20	0	71.6	72.3	3.1
2	82	20	5	57.2	56.8	1.6
3	82	45	0	71.7	71.2	1.0
4	82	45	5	57.0	58.7	4.9
5	98+	20	0	76.1	76.0	1.5
6	98+	20	5	68.0	68.8	1.4
7	98+	45	0	77.1	76.9	1.1
8	98+	45	5	69.7	70.4	1.6

Finally, the data from the above trials was analized using the Dupont experimental method. As a result, we found an equation for the expected brightness ($\hat{Y}$) of a deinked pad. The equation is as follows;

$\hat{Y}=68.83 - 5.26X + 0.51Y + 4.04Z + 1.62XZ$

Where X is the deinking temperature, Y is the blending time and Z is the chemical concentration based on the weight of the paper sample, (5g = 100%). See appendix "A" for details of method.

Discussion XI.

The Dupont equation may be confusing because of the reversal of signs in the formula. The (-5.26X) indicates that increasing the temperature from 82°C to 98^{+}°C will result in a brightness increase of 5.26 percent. The addition of 5 grams of napthalene to the deinking emulsion, surprisingly will result in over 4 points loss in brightness. There was also a brightness loss of 1.62 percent caused by the interaction of higher temperature while using napthalene. Blending time had only a slight effect on the brightness. These results were expected and are corroborated by the results tabulated on table 1, page44. However, it was not expected that the 0 gram napthalene runs would produce a higher brightness sheet compared to the 5 gram runs. Instead, we expected to see an increased brightness while using 5 grams of napthalene, because of better ink particulate removal. We also expected less retention of dyes and or, ink pigments in the deinked pad. The combined effects of these undesirable components tended to lower the measured brightness. In light of these results, it is not certain how much, if any, the napthalene helped in removal of ink from this particular paper sample.

Several explainations for these results are suggested below.

1) Contamination of the 0% runs with napthalene. After the 0% runs were filtered and drying, it was noticed that a detectable odor of napthalene was present in each of them. Upon examination of the blender, we noticed that the napthalene had attached and softened the plastic blender base. Possibly this was a source of small amounts of napthalene in the 0% runs. Another important consideration is that every other run used napthalene. Therefore, each 0% run should have approximately the same amount of residual napthalene in them.

2) The napthalene, when used in higher concentrations, tended to remove dye or disperse ink pigments.

3) Increased blending time decreased the ink particle size, which may have been retained with the fiber.

4) Another problem may have been the type of paper used. It was observed that the paper printed with more penatrating ink films deinked less well. See experiments VI. and VIII.

5) The dispersing and deinking effects of the T.S.P.P. could not be measured or standardized in these experiments.

It's not known how low a concentration of napthalene would produce deinking results, however, experiment IX. indicates that less than 2 grams or 20% could be used.

Apparently the above factors tended to lower the brightness of the final deinked pulp. However, this was not the general case as can be seen by comparing sample pads from this experiment with those of experiments IX. and X. See pad samples at the end of this paper.

Results and Conclusions

It was found that napthalene could be used to deink waste paper. Either using a 100% solvent, or a water-napthalene emulsion system. The results were good to very good in all paper sample tested. (See Table I)

We found that both napthalene and paradichlorobenzene were very good ink solvents, at or above their respectible melting points. Although the solvent properties released certain dyes from the inks, it would be expected that these chemicals could be used successfully in a process similar to the one used by the Riverside Corporation. The unique property of sublimation may be incorporated in a recovery design for these compounds. Both are sublimed at ordinary pressures and temperatures. Therefore, it may be possible to recover the solvent using a stream of hot, moist air. Instead of using steam stripping for recovery, and having to worry about possible water contamination.

Generally, the additions of napthalene to the deinking solution enhanced particulate ink removal, via flotation. Deinked samples using the water and napthalene emulsion are represented at the end of this paper. Not only were ink particles removed, but plastics, hotmelts, stickies and coating latexes. Some of these floated and others formed a colloidal suspension. In either case the result was the same; to separate the undesirable material from the desireable pulp. Problems were encountered with the wettability of various grades of paper. Due to this, there was a wide range of yields from each of the different deinking experiments. The amount of napthalene needed to promote good flotation was not optimized during these experiments. It is known however, that less than 20% concentration based on the A.D. weight of

the test paper could be used effectively. See experiment IX. A mathematical analysis was used on the data of experiment XI, to relate two different levels of addition of, 1) The blending time, 2) The chemical concentration, and 3) The temperature of deinking, to the pad brightness. It was shown that the temperature had the greatest effect on increasing the pad brightness, by as much as 5%, followed by a loss in brightness of 4% with increasing chemical concentration, and finally, a slight brightness loss with increased blend time. It was also noticed that the emulsion deinking system worked best on inks that were more plastic-like in nature. Furthermore, we observed less ink removal from inks that were less polyimerized, and those that contained dyes. What is not known is the amount of effect on deinking that the TSPP had in relation to the napthalene.

Recomendations

Better control of fiber wettability and yield would be useful in determination of the potential for this deinking process. A stainless steel blender should be used in any further experimentation to prevent solvent damage like that which occured in the one quart, plastic based, Waring blender. Extensive experimentation should be done using either napthalene or paradichlorobenzene in a 100% solvent deinking system. A comparison between solvent recovery by steam or hot air could produce interesting results. Sublimation as a means for solvent recovery should be explored. It would be interesting to know more about the chemistry and the mechanisms behind the ability of these solvents to remove ink. Combination systems composed of a napthalene emulsion in a standard deinking solution also offers a wide range of possibilities. To save time, and cover a broad range of experiments one should consider further work using the Dupont Experimental Analysis as an aid.

Graph of Napthalene and Camphor Eutectic Melting Points

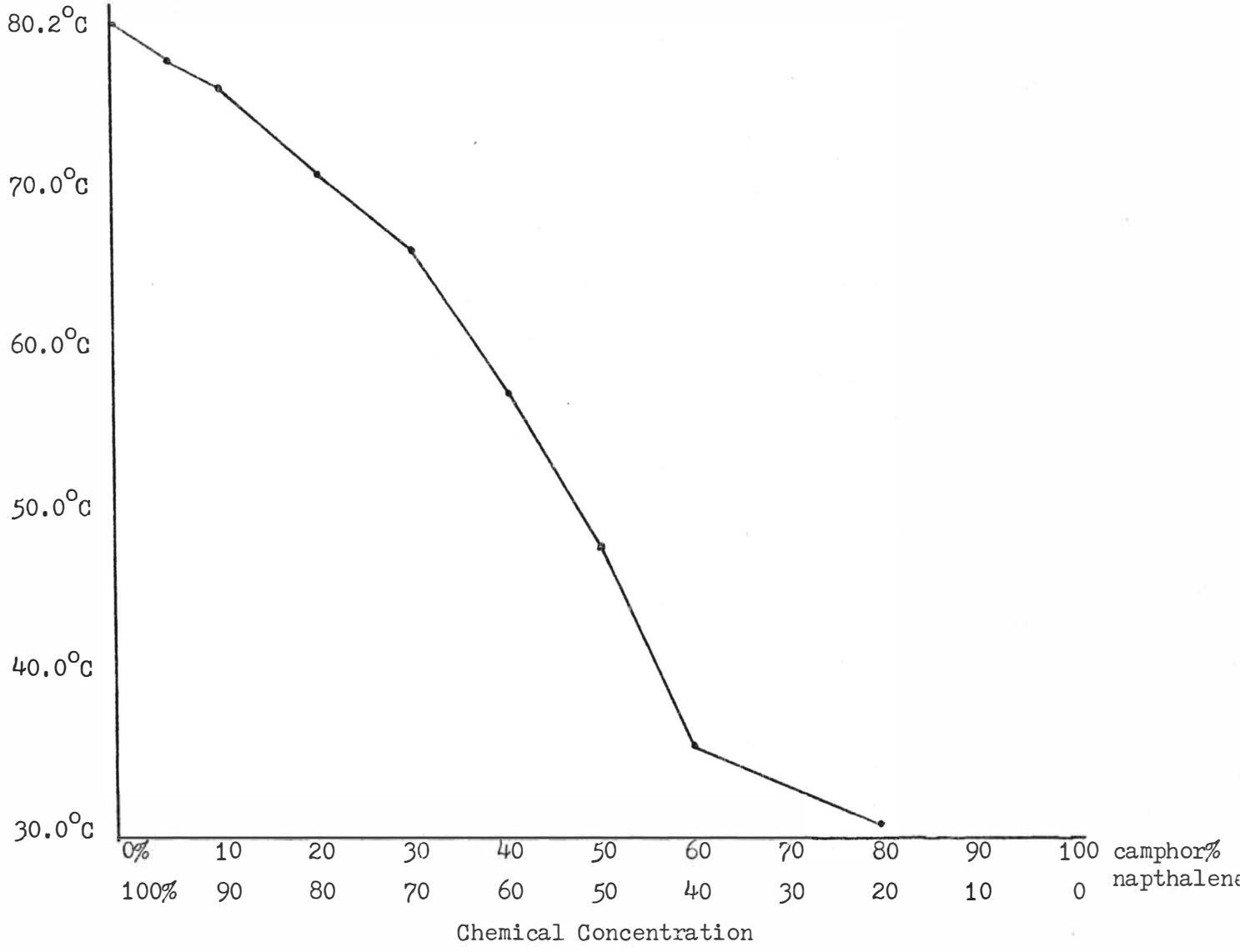

Fig. Graph of melting points of napthalene and camphor eutectic at various concentrations of each component.

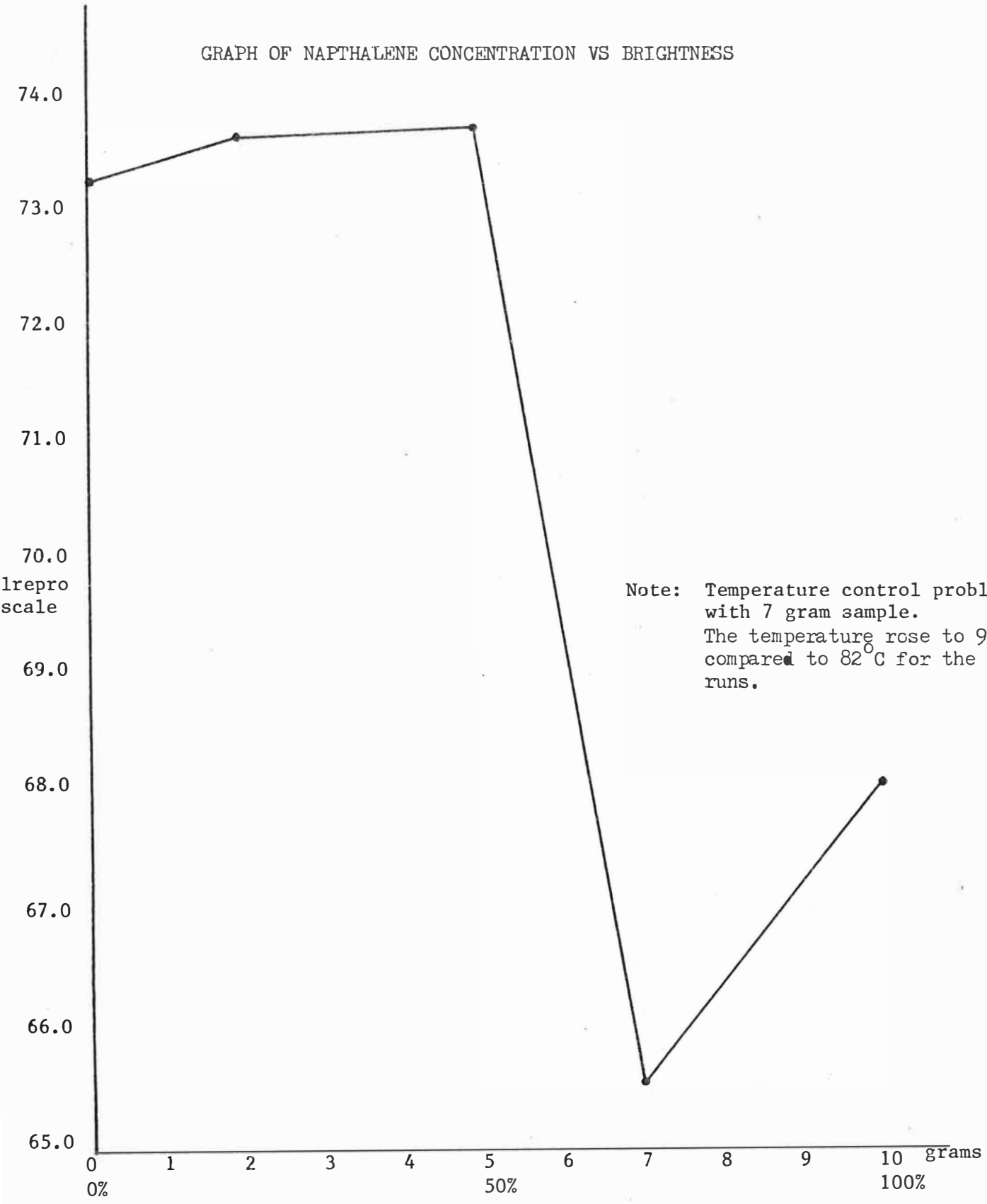

Fig. 2. Graph showing the Elrepro brightness vs the difference in chemical concentration based upon the weight of the unpulped sheet.

Napthalene Emulsion Deinking Results

Paper	Removal	Remarks	Yield%	Basestock Br.	Pad Br.
1) Plastic coated board "Munch Peanut bar box"	Very good	White sheet, no specks	26.8	77.2	74.0
2) Magazine cover "Home Center" magazine	Very good	Light purple tinge	39.4	79.8*	73.2
3) Uncoated, unfilled paper Neutral black ink	Good	Purple-grey color, uniform color	91.7	82.4	58.4
4) UV-cure inked box Sun Chemical Co. Kalamazoo	Very good	Slight yellow	37.4	83.9	75.2
5) Beer box Stroh's case	Very good	Usual color, no stickies	53.4	-----	-----
6) Old corrugating medium (hotmelts and stickies present)	Very good	Usual color, no stickies	80.3	-----	-----

*Note: This is the brightness of a coated sheet.

Table 1

APPENDIX "A"

Computation of Factor Effects and Predicted Linear Equation

TRIAL	MEAN ELREPRO	X	Y	XY	Z	XZ	YZ	XYZ
1	-71.88	-71.88	-71.88	+71.88	-71.88	+71.88	+71.88	-71.88
2	+56.99	+56.99	-56.99	-56.99	-56.99	-56.99	+56.99	+56.99
3	+71.46	-71.46	+71.46	-71.46	-71.46	+71.46	-71.46	+71.46
4	+58.85	+58.85	+58.85	+58.85	-58.85	-58.85	-58.85	-58.85
5	+76.02	-76.02	-76.02	+76.02	+76.02	-76.02	-76.02	+76.02
6	+68.40	+68.40	-68.40	-68.40	+68.40	+68.40	-68.40	-68.40
7	+77.00	-77.00	+77.00	-77.00	+77.00	-77.00	+77.00	-77.00
8	+70.05	+70.05	+70.05	+70.05	+70.05	+70.05	+70.05	+70.05
Sum of all (+)	550.65	254.29	277.36	276.80	291.47	281.79	275.92	274.52
Sum of all (-)	0	296.36	273.29	273.85	259.18	268.86	274.73	276.13
Overall sum	550.65	550.65	550.65	550.65	550.65	550.65	550.65	550.65
Difference	550.65	-42.07	+4.07	+2.95	+39.29	+12.93	+1.19	-1.61
Effects or (b-values	68.83	-10.52	+1.02	+0.74	+8.07	+3.23	+0.30	-0.403

Note: The next page shows the computation of the predicted Elrepro brightness line.

Computation of the Predicted Elrepro brightness line

1) Effect= $\frac{\text{Difference Value}}{\text{Number of (+) signs}}$ = (b-value)

2) Pooled Standard Deviation= $S_p = \left[\frac{(n_1 - 1)S_1^2 + (n_2 - 1)S_2^2 + \ldots (n_k - 1)S_k^2}{(n_1 - 1) + (n_2 - 1) + \ldots (n_k - 1)}\right]^{\frac{1}{2}}$

3) Minimum Effect= $t(S_p)\ (2/mk)^{\frac{1}{2}}$

4) Degrees of Freedom= 8+(4-1)=11 degrees

5) t-value for 11 D.F. (value of a table of values)= 2.20

6) m-value= number of (+) signs in a column

7) k-value= number of replications in a trial

1) $S_p = \left[\frac{(10-1)(0.688)+(10-1)(0.343)+(10-1)(0.112)+(10-1)(2.212)+(9)(0.273)+(9)(0.371)+(9)(0.149)(9)(0.505)}{9+9+9+9+9+9+9+9}\right]$

2) Minium Effect = (2.20)(0.763) (2/(4)(10)) = 0.375 Note: All significant values in Y are Min. effect value

3) Predicted Elrepro brightness equation = $\hat{Y} = \left[(68.83) - \frac{(10.52)X}{2} + \frac{1.02Y}{2} + \frac{0.74XY}{2} + \frac{8.07Z}{2} + \frac{3.23XZ}{2} + \frac{0.30YZ}{2} - \frac{0.403XYZ}{2}\right]$

$= 68.83 - 5.26X + 0.51Y + 4.04Z + 1.62XZ$

4) X= temperature (C^o)

Y= blend time (sec.)

Z= chemical concentration (0g = 0%,& 5g = 100%)

APPENDIX "A"

Table of Elrepro Brightnesses

TRIAL	ELREPRO BR. (Top of Pad)		ELREPRO BR. (Bottom of Pad)	
1	69.9,72.1,72.1,71.2,71.9;	$\bar{X}$=71.6	71.9,72.4,72.0,73.0,72.3;	$\bar{X}$=72.3
2	56.6,56.7,57.6,57.6,57.6;	$\bar{X}$=57.2	56.6,57.8,56.6,56.6,56.6;	$\bar{X}$=56.8
3	71.8,71.7,71.7,71.7,71.7;	$\bar{X}$=71.7	70.8,71.6,71.2,71.2,71.2;	$\bar{X}$=71.2
4	58.2,58.6,54.6,55.9,57.7;	$\bar{X}$=57.0	58.0,58.6,58.9,59.5,58.5;	$\bar{X}$=58.7
5	75.9,76.7,76.7,75.8,75.3;	$\bar{X}$=76.1	76.5,75.9,76.2,75.2,76.0;	$\bar{X}$=76.0
6	67.9,68.5,67.7,67.7,68.2;	$\bar{X}$=68.0	67.8,69.1,69.1,69.0,69.0;	$\bar{X}$=68.8
7	77.7,77.1,76.7,76.6,77.4;	$\bar{X}$=77.1	76.7,77.3,76.5,77.0,77.0;	$\bar{X}$=76.9
8	69.4,68.7,69.8,70.3,70.2;	$\bar{X}$=69.7	69.5,70.2,70.9,70.5,71.0;	$\bar{X}$=70.4

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See original paper for deinked samples mentioned in this paper. Original located in the Paper Engineering Department at Western Michigan University, Kalamazoo, Michigan.